

PROJECT NUMBER: 1752  
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke  
PROJECT LEADER: J. O. Lephardt  
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PERIOD COVERED: February, 1988

## I. CIGARETTE SMOKE INVESTIGATIONS

- A. Objective: Develop a method for the analysis of aldehydes in mainstream(MS) and sidestream(SS) cigarette smoke using 2,4 DNPH derivatization followed by separation and detection using an HPLC procedure.
- B. Results: The trapping and reaction conditions have been optimized. SS will be collected for 2 seconds at 5,20,35, and 50 seconds using one cigarette at 5 seconds, a second cigarette at 20 seconds, etc. for a total of four cigarettes per SS trap. MS will be collected from four cigarettes for each MS trap. Samples from the traps will be injected onto the HPLC for separation and quantitation of the aldehydes using appropriate 2,4 DNPH standards.
- C. Conclusions: The method for the SS analysis of selected aldehydes has been developed and easily can be applied to MS.
- D. Plans: Data will be collected to assess method reproducibility and comparisons will be made with data from other analytical techniques.
- E. References:
- E. Thomas, P.M. Notebook #8502, pp. 72-74.
- A. Objective: Develop a method for the real-time measurement of acrolein in MS and SS smoke using tunable diode laser(TDL) spectroscopy.
- B. Results: Experiments were performed and completed which showed that heated inlet lines were not necessary. The potential interference of relatively high concentrations of methanol was evaluated and conditions optimized to eliminate this effect. Using the acrolein permeation calibration of 57.5 ug/min., the SS delivery for Monitor 24 cigarettes (n=34) was 560+/-50ug/cigt (70+/-7ug/min.), and 0.84+/-0.07 mg/g of tobacco and paper consumed.
- C. Conclusions: The TDL method has been developed for MS and SS acrolein analyses(although not simultaneously) and SS data have been collected for the six circumference model cigarettes.
- D. Plans: MS acrolein deliveries will be determined for these selected models and provisions will begin for reactivating the MS and SS formaldehyde methods on the second TDL system.

**E. References:**

M. Parrish, P.M. Notebook #8548, p. 90.

- A. Objective:** Develop a method for the simultaneous determination of total ammonia in the MS and SS whole smoke for a single cigarette using ion chromatography (IC).
- B. Results:** Several experiments were performed to determine the distribution of total ammonia in the SS particulate and gaseous phases using differently designed impaction traps. Less than 2% of the total ammonia was detected in the SS TPM collected in the impaction traps and 98% was found in the gas phase. The established method was applied to the six circumference model cigarettes and the results confirmed the observations obtained from using the gaseous ammonia TDL procedure.
- C. Conclusions:** Sufficient data have been collected on a number of different cigarette samples to conclude that the total ammonia deliveries of SS whole smoke generally exceed by 10 to 20% the values obtained for SS gaseous ammonia using the TDL.
- D. Plans:** The MS/SS total ammonia whole smoke method using IC is now the method that will be used as needed. Priority will shift to developing methodology for quantitating benzene and toluene in MS and SS smoke.

**E. References:**

1. H. Randolph, P.M. Notebook #8475, p. 102.
2. Randolph, H., "The Simultaneous Determination of the Total Ammonia Delivery in MS and SS Smoke for Cigarette Models with 17, 20, and 24.8 mm Circumference," memo to J. Lephardt, February 3, 1988.

**II. ANALYTICAL SUPPORT TO R&D AND OTHER P.M. DEPARTMENTS**

- A. Objective:** Perform pyrolysis-GC/MS analyses as requested.

**B. Results:**

A sample of Angstrom Tech. Compound #5 was examined at 315 and 590°C. Six compounds were identified at 590°C.

The volatile pyrolysis products of a cigarette paper sample P8AB were compared to two control paper samples with no compounds observed in the P8AB sample that were not also observed in at least one of the control papers.

**C. References:**

1. Magin, D., "Pyrolysis-GC/MS of Angstrom Tech. Compound #5 U.V. Fluorescing Material," memo to R. Cox, February 5, 1988.

2. Magin, D., "Curie-Point Pyrolysis-GC/MS of Cigarette Paper P8AB," memo to R. Cox, February 11, 1988.

A. Objective: Perform GC/MS analyses as required.

B. Results:

A sample of a reaction mixture (#8177-199) from D. William was evaluated and it was determined that the expected compound was a minor component of the mixture.

C. Plans: Continue to provide pyrolysis-GC/MS and GC/MS support as needed and to initiate a systematic study of tobacco pectin.

A. Objective: To perform infrared qualitative analyses of commercial products prior to use in P.M. facilities as part of the Material Evaluation Program.

B. Results:

Samples of tube belts were identified as a combination of natural cellulose and polyethylene terephthalate fiber. Samples of conveyor belts were determined to have polyisoprene and polyester urethane.

Samples of machine parts submitted included a plastic cover of polyvinyl chloride, a white rubber sleeve of methyl silicone rubber, and a machine label of methyl/methacrylate.

C. Plans: Continue the analyses required in support of the Material Evaluation program.

D. References:

M. Griff, P.M. Notebook # 8554, pp. 53-59, 61-63.